



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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In re Application of

Group Art Unit: 1754

Teruhiko NAWATA, et al.

Examiner: Ngoc-Yen M. Nguyen

Serial No. 10/717,281

Filed: 11/19/2003

For: AS-GROWN SINGLE CRYSTAL OF ALKALINE EARTH METAL FLUORIDE

The Honorable Commissioner of Patents and Trademarks  
United States Patent and Trademark Office  
Washington, D. C. 20231

Sir:

DECLARATION UNDER 37 CFR 1.132

I, Teruhiko NAWATA, declare and state that:

1. In March, 1989, I was graduated from the graduate course of Kyushu University, Engineering Research Department, majoring in nuclear engineering and received a degree of Master of Engineering from the same University.

Since April, 1989, I have been an employee of TOKUYAMA CORPORATION, and till the present time I have been engaged in the research and development work concerning polymer science, polymer processing and crystal growth.

2. I am a co-inventor of the invention described in the specification of the above-identified application.

3. I carried out the following experiments.

#### Comparative Experiment I

Growing of a calcium fluoride single crystal was carried out using a crucible in which a pyrolytic carbon was deposited on the inner surface of a high-purity graphite crucible having an inside diameter of 60 mm and a height of 150 mm with the single crystal growth apparatus of the Bridgeman-Stockburger method (Bs method). Measurement of a surface of the crucible with the contact surface measuring instrument resulted in 2.1  $\mu\text{m}$  as an average roughness.

The resultant as-grown single crystal had a lateral surface comprising the very rough portion and the relative smooth portion. Measurement of a surface roughness of the relative smooth portion with the contact surface measuring instrument (manufactured by Taylor Hobson Ltd.) according to JIS B0651 resulted in 3.4  $\mu\text{m}$  as an average roughness. Measurement of a light transmittance of the as-grown single crystal at a wavelength of 632.8 nm resulted in 48.8%.

The resultant as-grown single crystal was subjected to the annealing treatment on condition according to First Preferred Embodiment of Sakuma et al (US6,332,922, see column 11, line 65 to column 12, line 39) with a sealable electric furnace for heat-treatment (heat-treating furnace). Specifically, the annealing-treatment was conducted in the following procedures.

The resultant as-grown single crystal was placed in a graphite container set in the heat-treating furnace. In addition, 1 g of zinc fluoride as a fluorination agent was placed in another graphite container, and then the container was set in the heat-treating furnace. The inside of the heat-treating furnace was vacuumed to  $1 \times 10^{-4}$  Pa by a vacuum pump, and then a temperature inside the heat-treating furnace was raised to 1050°C from ordinary temperature at a rate of 50°C/hour. Thereafter, high-purity argon gas was introduced into the heat-treating furnace to an atmosphere pressure, and then a temperature inside the heat-treating furnace was maintained at 1050°C for 24 hours. Thereafter, a temperature inside the heat-treating furnace was cooled to 900°C at a rate of 2°C/hour, and then was cooled to ordinary temperature from 900°C at a rate of 5°C/hour.

Measurement of a surface roughness of the relative smooth portion of the resultant single crystal with the contact surface measuring instrument (manufactured by Taylor Hobson Ltd.) according to JIS B0651 resulted in 3.0  $\mu\text{m}$  as an average roughness. Measurement of a light transmittance of the heat-treated single crystal at a wavelength of 632.8 nm resulted in 47.2%.

Accordingly, a light transmittance of the as-grown single crystal was not improved by the annealing treatment.

## Comparative Experiment II

Pulling of a calcium fluoride single crystal was carried out in the same manner as in Example 1 of the specification, except that in the single crystal pulling apparatus of Fig. 1, the barrier (13) was not provided. As a result, an as-grown single crystal of calcium fluoride having a straight barrel part maximum diameter of 28 cm and a weight of 16.5 kg was prepared. The length of the straight barrel part of the as-grown single crystal was 6 cm.

Measurement of a surface roughness of the as-grown single crystal with the contact surface measuring instrument (manufactured by Taylor Hobson Ltd.) according to JIS B0651 resulted in  $0.92\text{ }\mu\text{m}$  as an average roughness. Measurement of a light transmittance of the as-grown single crystal at a wavelength of 632.8 nm resulted in 72.5%.

The resultant as-grown single crystal was subjected to the annealing treatment in the same manner as in Comparative Experiment I. Measurement of a surface roughness of the relative smooth portion of the resultant single crystal with the contact surface measuring instrument (manufactured by Taylor Hobson Ltd.) according to JIS B0651 resulted in  $0.89\text{ }\mu\text{m}$  as an average roughness. Measurement of a light transmittance of the heat-treated single crystal at a wavelength of 632.8 nm resulted in 73.4%.

Accordingly, a light transmittance of the as-grown single crystal was not improved by the annealing treatment.

The undersigned declares further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Respectfully submitted,

Teruhiko Nawata

Teruhiko NAWATA

This 29th day of May, 2007